CANADA
DEPARTMENT OF ENERGY, MINES AND RESOURCES
MINES BRANCH
OTTAWA

PHYSICAL METALLURGY DIVISION
INTERNAL REPORT PM-R-73-13

A STUDY OF THE PROPERTIES AND SINTERING BEHAVIOUR OF CERTAIN NICKEL POWDERS

Progress Report No. 5

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C. F. Dixon and H. M. Skelly

November 20, 1973

90-04664/

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ABSTRACT

The sintering behaviour at 1100°C (2012°F) of four nickel powders and two series of blends was investigated. Three levels of compacting pressure and three sintering times were employed. Evaluation of the sintered compacts was carried out by transverse rupture and tensile testing, density and porosity determination, and microexamination.

As in the case of earlier work in which a sintering temperature of 900°C (1650°F) was employed, highest strength and greatest response to sintering was shown by a fine (~6 μ) dendritic powder when compared to a range of fine and coarse spheroidal powders.

Density was influenced more by compacting pressure than by sintering time, and porosity remained mainly interconnected even after 2 hr of sintering.

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1. INTRODUCTION

This is the final report in a series describing the results of a study of the particle morphology and sintering behaviour of a variety of nickel powders and blends. Earlier reports[1-4] have dealt with the properties of the non-compacted powders and blends and their behaviour on compacting and sintering. This report provides more data on sintering behaviour. The last report[4] dealt with sintering at 900°C (1650°F), whereas a temperature of 1100°C (2012°F) was employed in the work reported here.

2. POWDERS AND BLENDS

The same powders and blends described in earlier reports[1-4] were used in this work. The four nickel powders were:

Powder Grade	Morphology	Particle Size, microns
Sherritt Gordon "S"	Spheroidal, pebbly	149 to <44
" " -100 +140S	" "	<149 to >105
" " SF 300	" "	~19
" " G-29	Loose, dendritic agglomerates	~ 6

The six blends were SF300 plus 25, 50 and 75% G-29, and -100 +140S with the same amounts of G-29.

3. EXPERIMENTAL PROCEDURE

3.1 Compacting and Sintering

Compacting and sintering conditions have already been described[4]. Briefly, transverse rupture and tensile test specimens were prepared at compacting pressures of 42.7, 64, and 96 ksi (295, 442, and 662 MN/m^2) for the former and 53.3, 66.7, and 80 ksi (368, 460, and 552 MN/m^2) for the latter. No lubricant was added to the powders, but the die was lubricated with stearic acid. The compacts were sintered in hydrogen at 1100°C (2012°F) for ½, 1, and 2 hr for each compacting pressure.

3.2 Mechanical Testing

Testing was conducted on an Instron machine. The transverse rupture specimens measured 1.25 x 0.5 x 0.25 in. (32 x 13 x 6 mm) (MPIF Standard 13-62). The tensile specimens were 3.5 in. (89 mm) long x 0.2 x 0.25 in. (5 x 6 mm) at the gauge section (MPIF Standard 10-63).

3.3 Density and Porosity

Density and porosity were determined for specimens corresponding to those used previously[4]. The specimen is impregnated with oil under vacuum and weighed in air and water; the volume of oil absorbed by the sample represents interconnected porosity (MPIF Standard 8-50).

3.4 Microexamination

Microexamination was carried out on sintered specimens which were impregnated and mounted in an epoxy resin prior to polishing and etching with Carapella's Reagent.

4. RESULTS AND DISCUSSION

4.1 Mechanical Properties

The transverse rupture test results are listed in Table 1, and representative results are plotted in Figures 1 to 3.

Figures 1 and 2 are plots of transverse rupture strength against sintering time for 3 of the powders (G-29, SF 300 and -100 +140S) and their blends, compacted at 64 ksi (442 MN/m^2). For comparison, the plot for the "standard" powder, S, is also included in Figures 1 and 2. These plots show that transverse rupture strength increases with increasing G-29 content. Also, the higher the G-29 content the more the strength is affected by sintering time. The apparent decrease in the strength of G-29 with increasing sintering time is thought to arise from scatter in the test results. There is considerable shrinkage of G-29 compacts on sintering, leading to warpage, and this could affect mechanical testing.

Comparison of the results plotted in Figures 1 and 2 with plots for 900°C (1650°F) sintering shows them to be similar, except that as G-29 content increases the sinterability increases more at the higher temperature.

The effect of compacting pressure on transverse rupture strength for G-29 and -100 +140S is shown in Figure 3. Similar plots for the other powders and blends would fall between those in Figure 3. The results indicate the slight effect sintering time and compacting pressure have on the strength of the -100 +140S powder. These results are similar to those obtained for specimens sintered at 900°C (1650°F)[4]. The scatter in the G-29 results in Figure 3 does not clearly indicate any significant effect from sintering time and compacting pressure as was the case for specimens sintered at 900°C (1650°F)[4].

The ultimate tensile and yield strengths and the elongation are given in Tables 2 to 4. Representative plots for the ultimate tensile results are given in Figures 4 to 6.

Figures 4 and 5 show how the tensile strength increases with increasing additions of G-29 to -100 +140S or SF300. It is also noticeable that the rate of increase in strength decreases as sintering time increases.

Figure 6 shows the effect of compacting pressure on sintered tensile strength for G-29 and -100 +140S. The plots for SF300 and the blends would come between these two sets of plots. The effect of increased compacting pressure on the sintered strength of -100 +140S is slight. There is no marked regular trend in the plots for G-29 and it is possible that warpage of the compacts during sintering resulted in some scatter.

The results for sintering at 1100°C (2012°F) are generally similar to those obtained for sintering at 900°C (1650°F). The strengths are higher, especially for G-29 and blends with high content of G-29, after sintering at 1100°C (2012°F) as compared with 900°C (1650°F).

4.2 Density and Porosity

Table 5 lists the density and porosity of representative specimens. As well as "g/cm^3", density is shown as a percentage of "theoretical" density (% TD), taking the density of solid nickel to be 8.90 g/cm^3. Total porosity, expressed as a percentage of specimen volume, was calculated from the relationship:

$$\% \text{ total porosity} = 100 - \% \text{ theoretical density}$$

Increasing the sintering time from ½ to 2 hr produced a small but significant increase in the density of -100 +140S. When the powder was sintered at 900°C (1650°F) there was no change in density on increasing the sintering time from ½ to 2 hr[4]. Increasing the compacting pressure produced a considerably larger increase in density. The same remarks apply to the results for the blend of -100 +140S with 50% G-29. Except for -100 +140S compacted at 42.7 ksi (295 MN/m^2) the density values were higher for the 1100°C (2012°F) sinter as compared to the 900°C (1650°F) sinter reported previously[4].

Porosity decreases with increasing sintering time and increasing compacting pressure for both -100 +140S and the 50% blend with G-29. As in the case of sintering at 900°C (1650°F)[4], the porosity in all cases is mainly interconnected.

4.3 Microstructure

The microstructures were similar to those of specimens sintered at 900°C (1650°F). The main difference was that the higher sintering temperature induced more internal voids within the powder particles of -100 +140S after ½ hr of sintering time; this effect can be seen by comparing Figure 7 (from this work) with Figure 8[4].

5. CONCLUSIONS

The conclusions listed in the report on similar work carried out at the lower sintering temperature of 900°C (1650°F)[4], generally apply to the present study. The order of sintered strength remains unchanged, increasing from coarse, spheroidal powder (-100 +140S) to fine, dendritic (G-29). The dendritic powder was more responsive to sintering time and compacting pressure than the spheroidal powders, and additions of the dendritic powder to the spheroidal powders increased the strength of the latter.

Density of the sintered compacts increased slightly with longer sintering times and there was an appreciable increase in density with increasing compacting pressure. Porosity was largely interconnected even after 2 hr of sintering.

The tensile and transverse rupture strengths for the 1100°C (2012°F) sinter were higher than those obtained for the 900°C (1650°F) sinter[4].

The development of voids within the coarse spheroidal powder particles during sintering was more noticeable after the ½ hr sinter at 1100°C (2012°F) than at 900°C (1650°F).

6. ACKNOWLEDGEMENT

Much of the laboratory work was performed by Mr. J. McGoey, Technical Assistant in this Section.

77. REFERENCES

1. H. M. Skelly and C. F. Dixon. "A Study of the Properties and Sintering Behaviour of Certain Nickel Powders, Progress Report No. 1", Physical Metallurgy Division Internal Report PM-R-68-9, Mines Branch, Department of Energy, Mines and Resources, Ottawa (April 23, 1968).

2. H. M. Skelly and C. F. Dixon. "A Study of the Properties and Sintering Behaviour of Certain Nickel Powders, Progress Report No. 2", Physical Metallurgy Division Internal Report PM-R-68-18, Mines Branch, Department of Energy, Mines and Resources, Ottawa (Nov. 5, 1968).

3. H. M. Skelly and C. F. Dixon. "A Study of the Properties and Sintering Behaviour of Certain Nickel Powders, Progress Report No. 3", Physical Metallurgy Division Internal Report PM-R-71-6, Mines Branch, Department of Energy, Mines and Resources, Ottawa (April 30, 1971).

4. C. F. Dixon and H. M. Skelly. "A Study of the Properties and Sintering Behaviour of Certain Nickel Powders, Progress Report No. 4", Physical Metallurgy Division Internal Report PM-R-71-10, Mines Branch, Department of Energy, Mines and Resources, Ottawa (Aug. 12, 1971).

TABLE 1

Transverse Rupture Strength of Sintered Compacts

Sintering Temperature: 1100°C (2012°F)

Compacting Pressure ksi (MN/m^2)	Sintering Time (hr)	Transverse Rupture Strength (ksi)									
						-100 +140S with:			SF300 with:		
		S	-100 +140 S	SF300	G-29	25% G-29	50% G-29	75% G-29	25% G-29	50% G-29	75% G-29
42.7 (295)	½	4.51	1.83	9.08	49.18	12.60	25.56	39.66	20.34	34.92	44.24
	1	6.47	2.69	12.27	61.86	13.03	31.72	49.34	22.39	39.68	54.79
	2	8.03	3.14	17.68	NF	23.92	43.13	61.47	34.21	56.65	NF
64.0 (442)	½	8.71	4.90	16.22	60.50	17.83	32.49	48.69	27.63	40.87	53.24
	1	11.40	5.54	18.99	76.26	20.58	37.05	56.71	33.13	51.90	63.98
	2	16.13	7.36	26.70	69.36	24.91	54.98	NF	40.30	NF	NF
96.0 (662)	½	16.56	7.81	26.00	77.59	26.12	41.39	58.82	40.48	54.32	65.49
	1	18.15	9.24	32.98	63.88	30.55	47.82	73.12	41.63	65.24	75.66
	2	20.55	12.88	45.13	70.68	40.32	66.07	NF	NF	NF	78.38

NF: no fracture

Note: in Tables 1 to 3, the equivalent MN/m^2 values can be obtained by multiplying the ksi figures by 6.9.

TABLE 2

Tensile Strength of Sintered Compacts

Sintering Temperature: 1100°C (2012°F)

Compacting Pressure ksi (MN/m^2)	Sintering Time (hr)	Ultimate Tensile Strength (ksi)									
						-100 +140S with:			SF300 with:		
		S	-100 +140 S	SF300	G-29	25% G-29	50% G-29	75% G-29	25% G-29	50% G-29	75% G-29
53.3 (368)	½	2.37	1.68	6.42	29.75	5.01	12.93	20.92	10.70	17.33	21.30
	1	3.77	1.07	8.18	38.45	8.70	20.73	30.00	14.67	24.10	31.95
	2	4.55	2.25	10.04	41.35	7.68	22.30	34.00	17.90	26.20	36.60
66.7 (460)	½	2.97	1.32	8.52	31.70	7.80	17.05	25.05	12.91	18.12	24.95
	1	4.18	2.88	11.93	37.70	10.74	23.13	31.15	17.78	26.80	33.25
	2	4.56	3.31	13.40	35.90	9.48	24.05	36.40	21.85	30.32	38.50
80.0 (552)	½	3.90	2.54	9.83	39.10	7.49	17.50	27.30	14.60	22.65	26.85
	1	4.46	3.16	13.93	39.60	11.16	23.50	33.40	18.50	27.60	38.70
	2	5.68	3.82	14.85	35.00	14.10	28.20	37.50	23.80	34.90	40.60

TABLE 3

Yield Strength of Sintered Compacts

Sintering Temperature: 1100°C (2012°F)

Compacting Pressure ksi (MN/m^2)	Sintering Time (hr)	Yield Strength (0.2% offset) ksi									
						-100 +140S with			SF300 with		
		S	-100 +140 S	SF300	G-29	25% G-29	50% G-29	75% G-29	25% G-29	50% G-29	75% G-29
53.3 (368)	½	1.93	1.31	3.76	15.43	5.01	8.91	11.42	6.73	10.26	13.15
	1	2.12	ND	3.82	14.20	4.78	8.51	11.78	6.95	10.37	12.93
	2	2.03	1.15	3.89	13.40	5.0	7.87	10.83	6.86	10.09	12.0
66.7 (460)	½	2.28	1.28	4.78	15.83	5.98	9.14	12.41	7.79	10.54	13.48
	1	2.14	1.44	4.52	14.62	5.48	8.70	12.05	7.45	10.97	13.25
	2	2.23	1.53	4.58	12.25	5.30	8.19	10.99	7.48	10.20	11.92
80.0 (552)	½	2.78	2.03	5.52	14.96	5.94	9.60	12.96	8.45	11.65	13.98
	1	2.53	1.65	5.06	14.48	5.51	8.78	12.28	8.15	11.02	12.80
	2	2.63	1.70	4.82	12.25	5.36	8.64	11.0	7.72	10.48	11.50

TABLE 4

Elongation of Sintered Compacts

Sintering Temperature: 1100°C (2012°F)

Compacting Pressure ksi (MN/m^2)	Sintering Time (hr)	Elongation (% on 1 in.)									
						-100 +140S with:			SF300 with:		
		S	-100 +140 S	SF300	G-29	25% G-29	50% G-29	75% G-29	25% G-29	50% G-29	75% G-29
53.3 (368)	½	2	2	5	9.5	2	4	6	4	6	5
	1	2	1	5	18	3.5	8	11	8	8	14
	2	4	3	10	21	4.5	10	15	10	11	17
66.7 (460)	½	2	1	5	10	2	6	8	5	5	7
	1	3	2	9	16	5.5	9	12	9	10	12
	2	3	4	11	12	2.5	10	18	12	13	20
80.0 (552)	½	2	1	5	12	2	6	9	5	7	8
	1	1	3	10	16.5	3.5	9	12	7	10	21
	2	5	3	11	12	4.5	11	18	12	18	22

TABLE 5

Density and Porosity of Specimens Sintered

at 1100°C (2012°F)

Powder or Blend	Compacting Pressure ksi (MN/m^2)	Sintering Time (hr)	Density		Porosity, %	
			g/cm^3	% TD*	Connected	Total
-100 +140S	42.7 (295)	½	5.27	59.2	40.5	40.8
"	"	2	5.52	62.0	35.2	38.0
"	96 (662)	½	6.37	71.6	25.2	28.4
"	"	2	6.58	73.9	24.3	26.1
-100 +140S with 50% G-29	42.7 (295)	½	6.92	77.7	20.5	22.3
"	"	2	7.19	80.8	17.8	19.2
"	96 (662)	½	7.59	85.3	15.2	14.7
"	"	2	7.70	86.5	10.9	13.5

*taking density of nickel as 8.90 g/cm^3

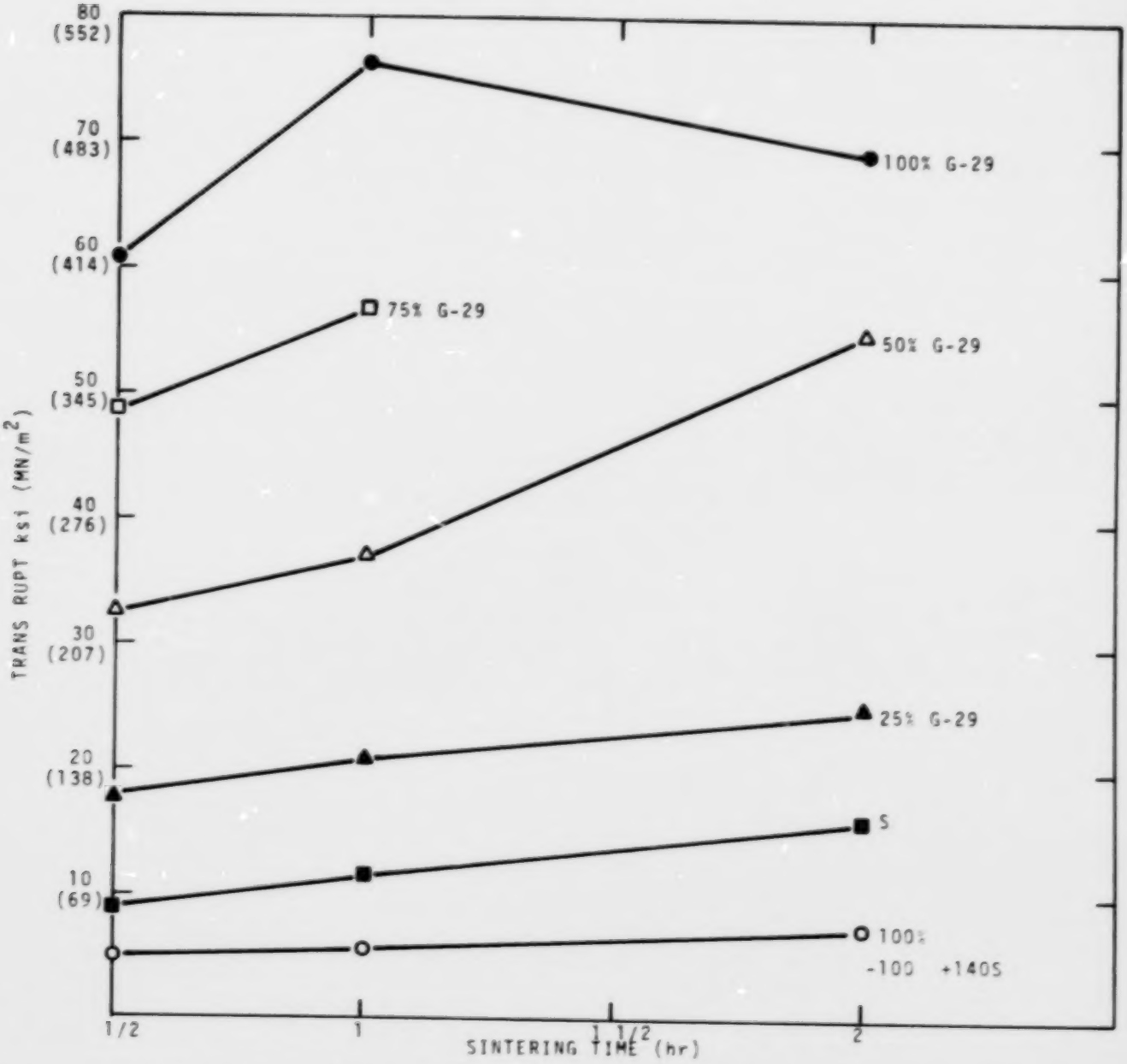

FIGURE 1. Trans Rupt vs Sintering Time for G-29, -100 +140S and Blends. Compacted at 64 ksi (442 MN/m²). Sintered at 1100°C (2012°F)

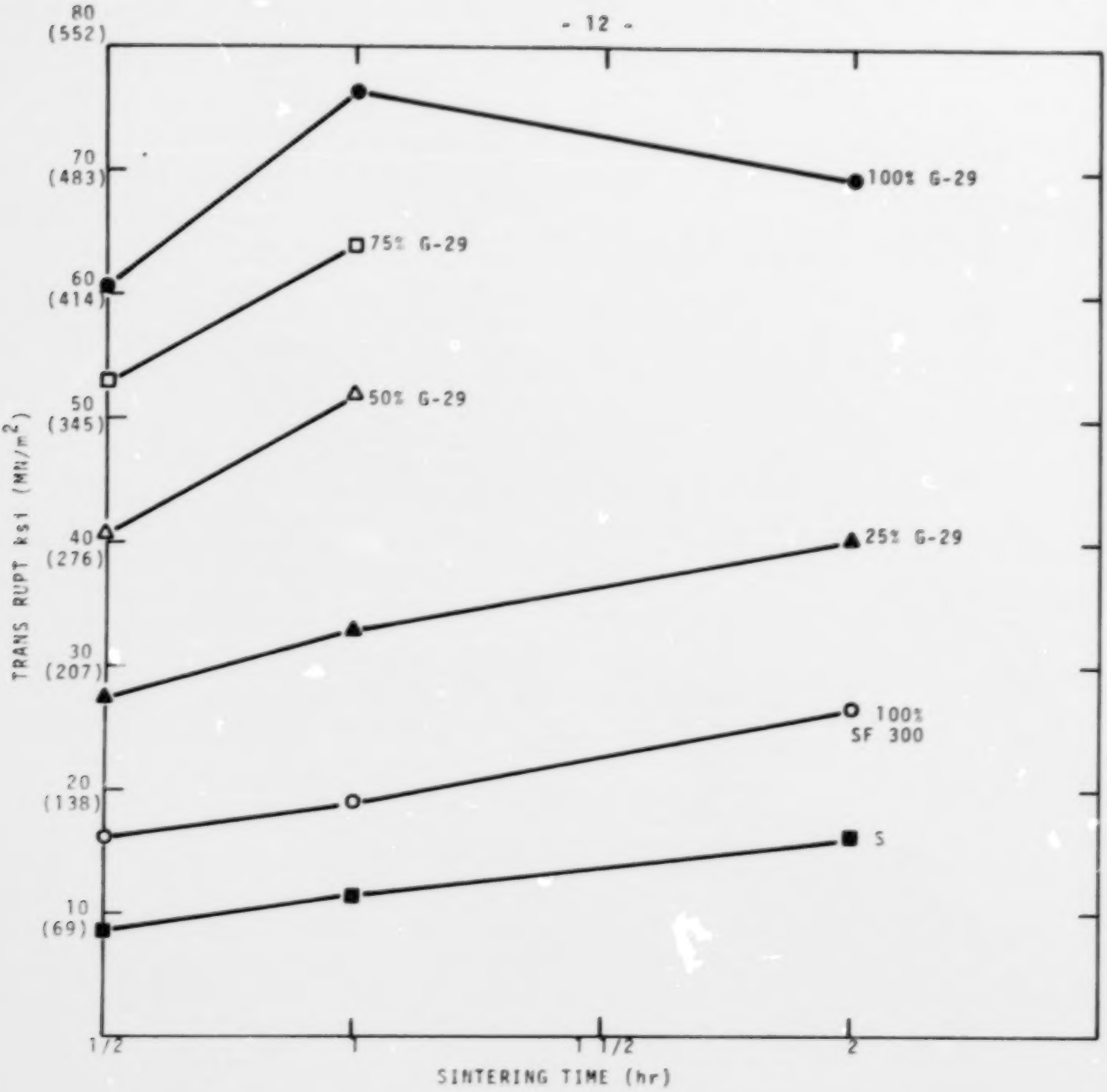

FIGURE 2. Trans Rupt vs Sintering Time for G-29, SF300 and Blends. Compacted at 64 ksi (442 MN/m^2). Sintered at 1100°C (2012°F).

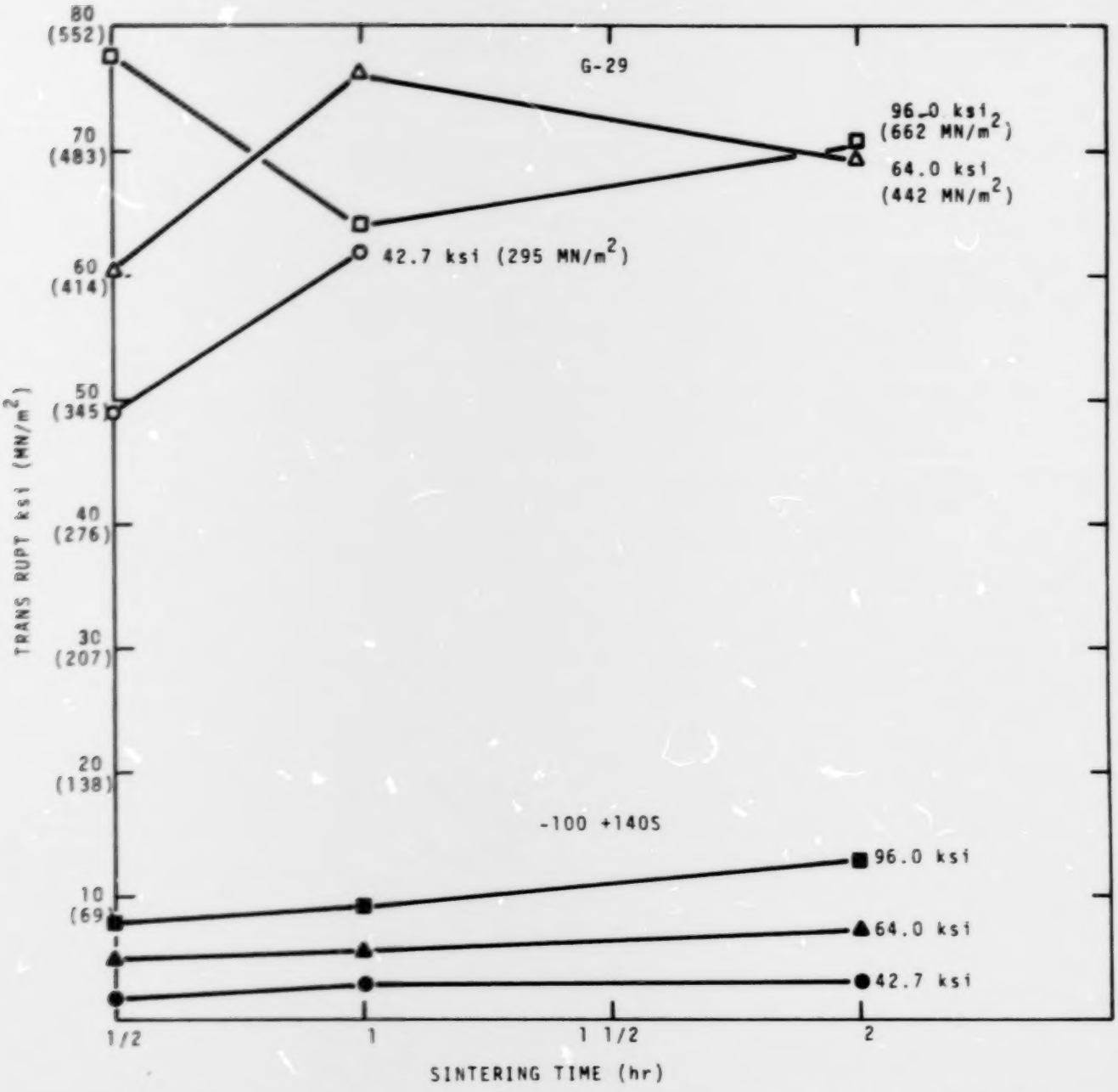

FIGURE 3. Trans Rupt vs Sintering Time for G-29 and -100 +140S at Various Compacting Pressures. Sintered at 1100°C (2012°F).

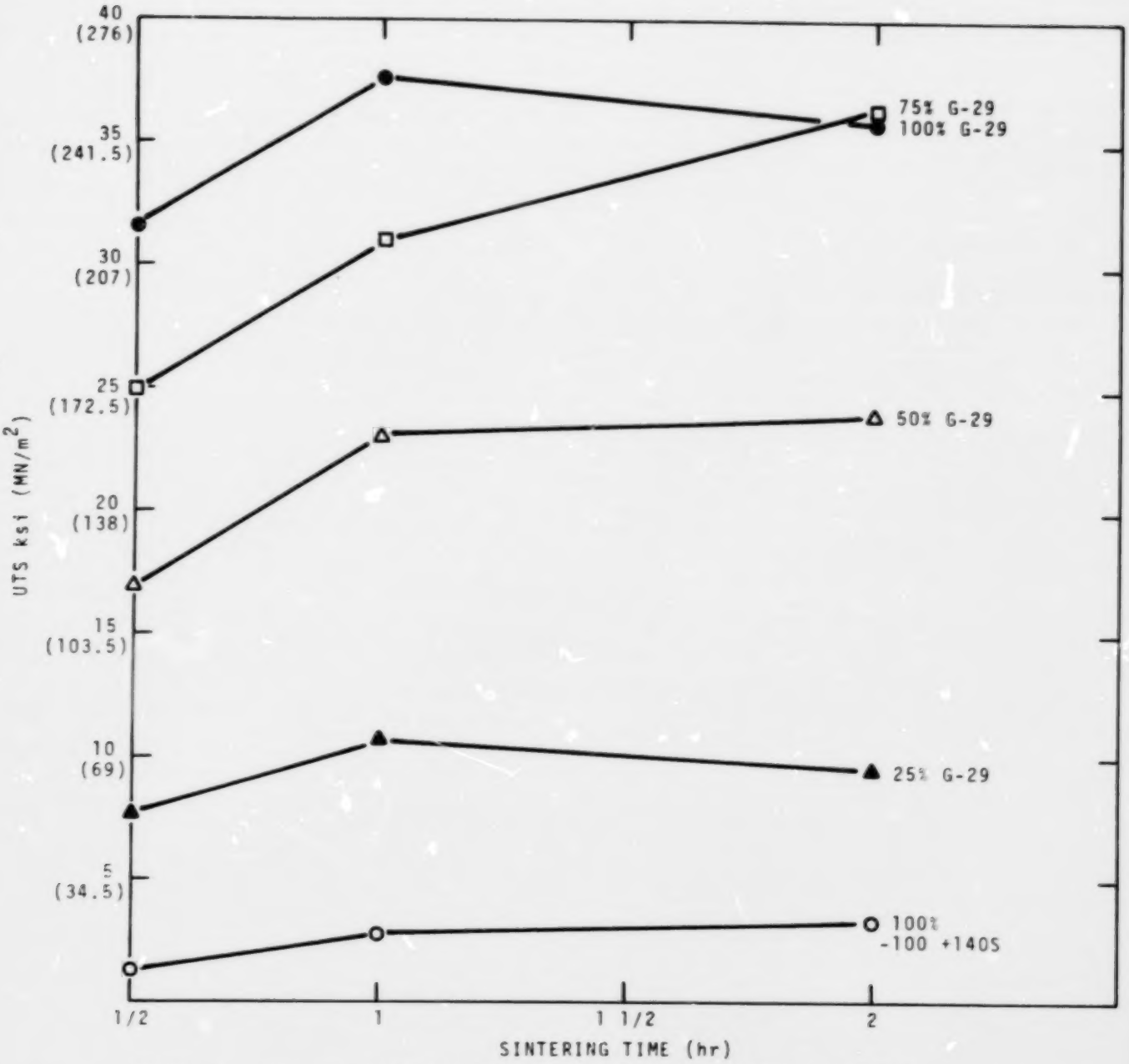

FIGURE 4. UTS vs Sintering Time for G-29,$_2$-100 +140S and Blends. Compacted at 66.7 ksi (460 MN/m^2). Sintered at 1100°C (2012°F).

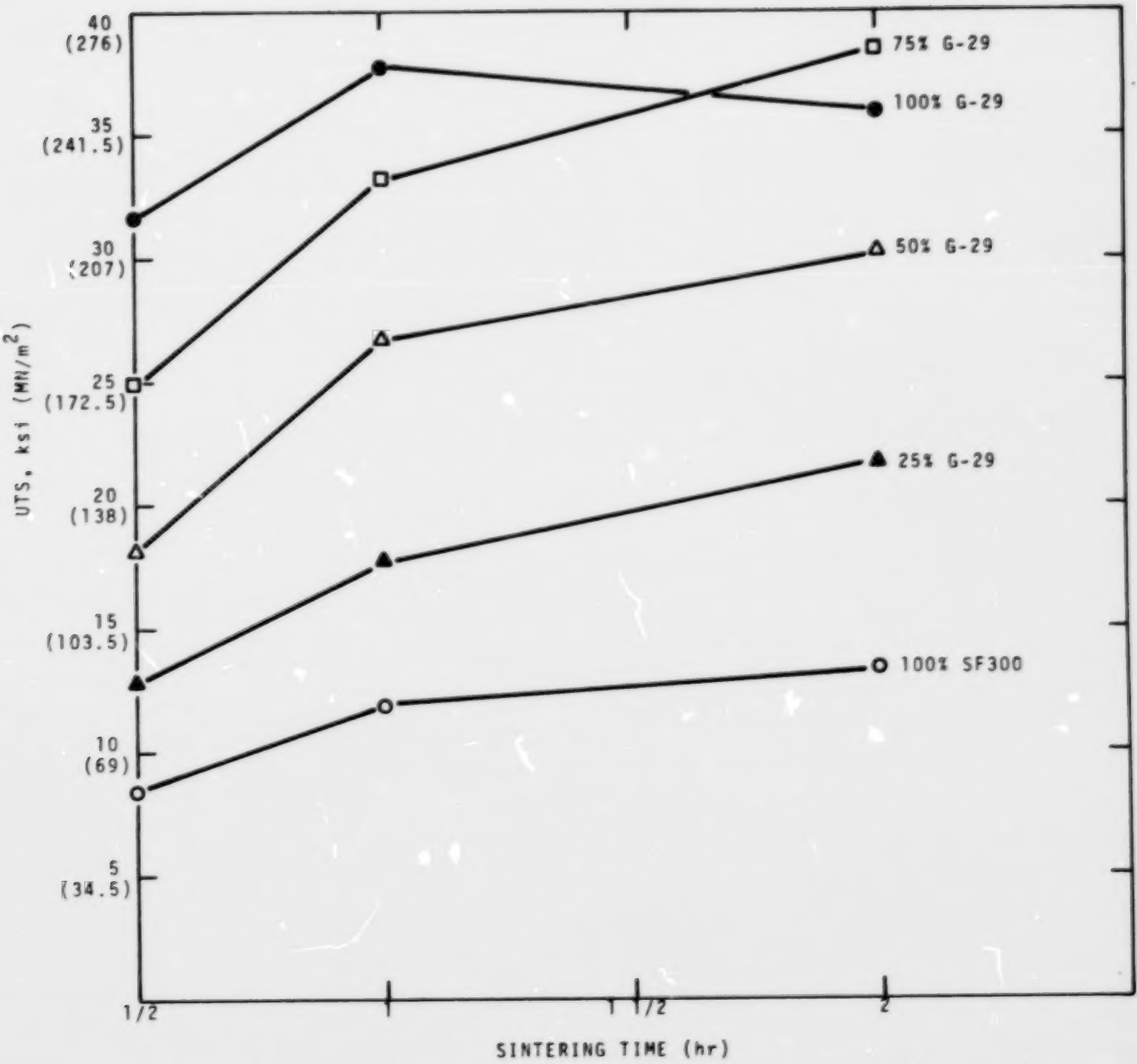

FIGURE 5. UTS vs Sintering Time$_2$ for G-29, SF300 and Blends. Compacted at 66.7 ksi (460 MN/m^2). Sintered at 1100°C (2012°F).

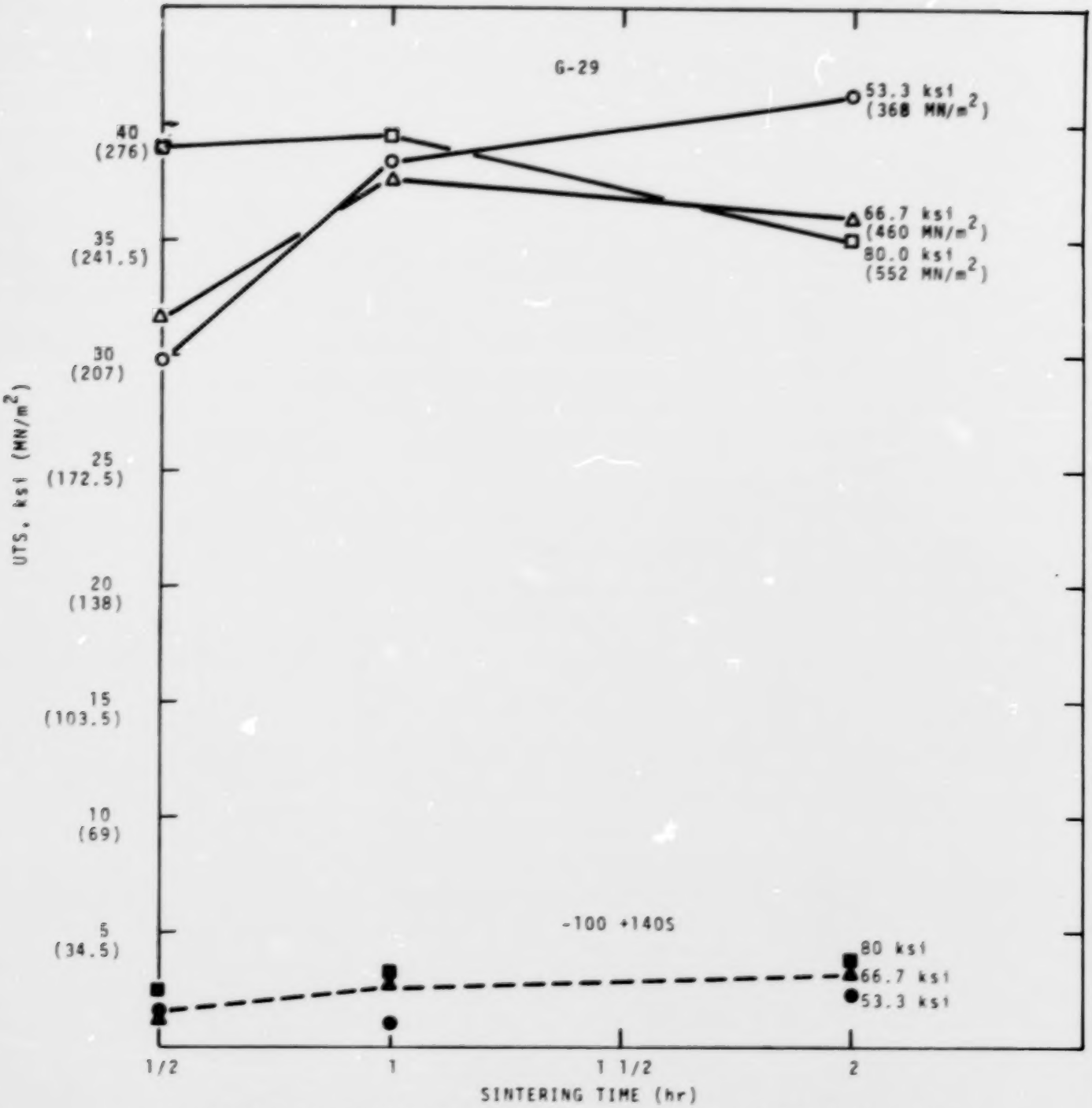

FIGURE 6. UTS vs Sintering Time for G-29 and -100 +140S at Various Compacting Pressures. Sintered at 1100°C (2012°F).

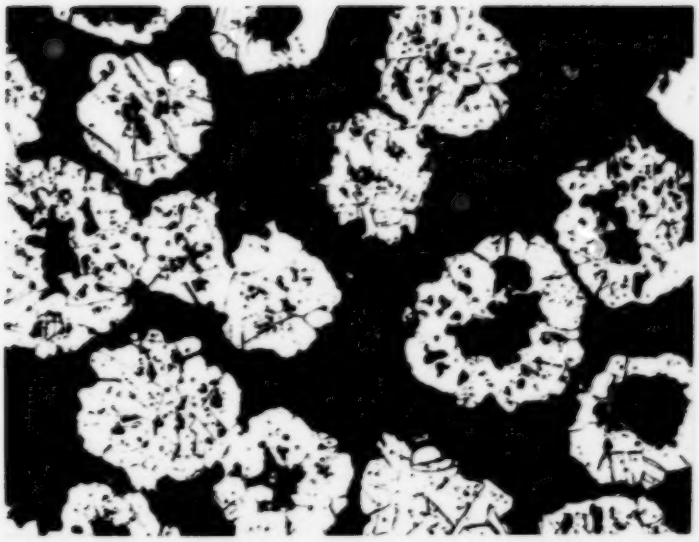

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Figure 7. Powder grade -100 +140S compacted at 42.7 ksi (295 MN/m^2) and sintered at 1100°C (2012°F) for ½ hr.

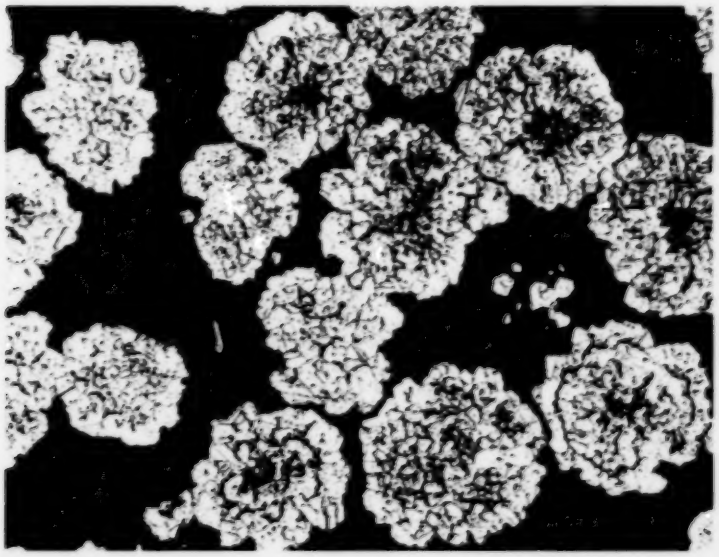

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Figure 8. Powder grade -100 +140S compacted at 42.7 ksi (295 MN/m^2) and sintered at 900°C (1650°F) for ½ hr.